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SYNTHESIS

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# New!

# Phase-Transfer Reactions

## Fluka-Compendium Volume 1

By **Walter E. Keller**
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Phase-transfer catalysis and related methods have emerged as preparative tools of prime importance. As techniques of conducting reactions in a multi-phase system, these methods have been applied successfully to a wide range of organic reactions. Results of these applications have led to more than two thousand papers published in the last twenty years on the subject, inciuding numerous monographs and reviews.
Not surprisingly, hundreds of patents have appeared in the past, showing that the method of phase-transfer reaction has also found its application in industrial-scale processes.

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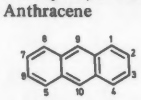

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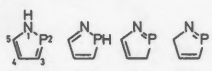

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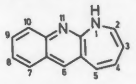

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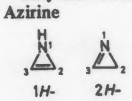

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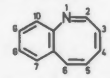

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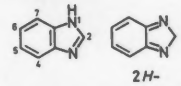

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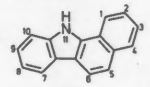

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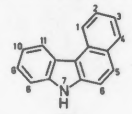
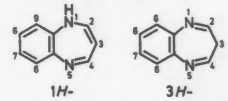


7*H*-

1,5-Benzodiazepine



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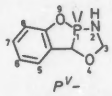
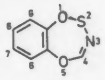


$P^V$–

1,5,2,3-Benzodioxathiazepine



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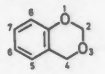
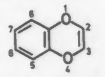


1,4-Benzodioxin



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1,4-Benzodioxins, 2-Silyl- 337
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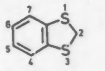

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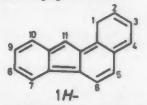
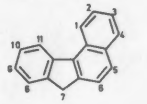


1*H*-

11*H*-Benzo[*a*]fluorenes 366
11*H*-Benzo[*a*]fluorene-11-carboxylic Esters 366
- Dealkoxycarbonylation 366
Benzo[*c*]fluorene



7*H*-

7*H*-Benzo[*c*]fluorenes 366
7*H*-Benzo[*c*]fluorene-7-carboxylic Esters 366
- Dealkoxycarbonylation 366
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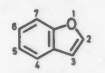

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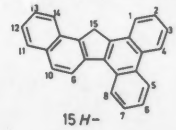

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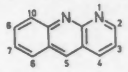
Benzo[*b*][1,8]naphthyridine



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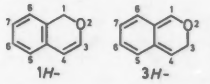

1*H*- 3*H*-

Isochromans, 1-(2-Oxocycloalkyl)- 1004
Isocoumarins 944
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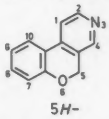
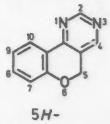


5*H*-

5-Oxo-5*H*- 136
[1]Benzopyrano[4,3-*d*]pyrimidine



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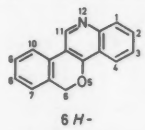
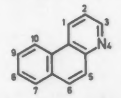
3,4-Dihydro-5*H*-, 2-(2-Hydroxyphenyl)-4,5-dioxo- 147
3,4-Dihydro-5*H*-, 2-(2-Hydroxyphenyl)-5(4)-imino-4(5)-oxo- and 5-Imino-4-thioxo- 147
3,4-Dihydro-5*H*-, 2-(2-Hydroxyphenyl)-5-oxo-4-thioxo- 147
[2]Benzopyrano[4,3-*c*]quinoline



6*H*-

6-Oxo-6*H*- 76, 1123
Benzo[*f*]quinoline



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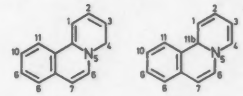
Benzo[*a*]quinolizine



4*H*- 11b*H*-

5,6-Dihydro-8*H*-, 8-Dicyanomethylene- 908
6,7-Dihydro-4*H*-, 4-Diacylmethylene-, 4-Dicyanomethylene-, and 4-Oxo- 908
2,3,6,7-Tetrahydro-4*H*-, 4-Diacylmethylene- 908

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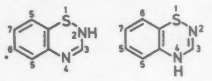
5,6,9,10,11,11a-Hexahydro-8*H*- 908
**1,2,4-Benzothiadiazine**



2*H*- 4*H*-

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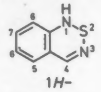
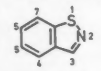


1*H*-

1*H*-2,1,3-Benzothiadiazine 2,2-Dioxides 440
**1,2-Benzothiazole**



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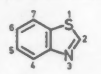
**1,3-Benzothiazole**



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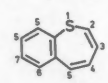
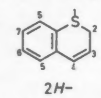
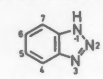


2,3-Dihydro-1-benzothiepins, 4-Acyl--5-aryl- 744
**1-Benzothiopyran**



2*H*-

2*H*-1-Benzothiopyrans, 3-Acyl-4-aryl- 744
**Benzotriazole**



Benzotriazoles, 2-(2-Hydroxyphenyl)- 647
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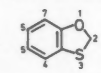

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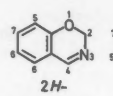

2*H*- 4*H*-

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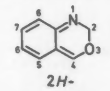
**3,1-Benzoxazine**



2*H*- 4*H*-

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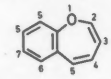

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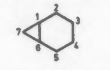

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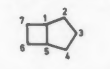

Bicyclo[3.2.0]hept-2-enes, 2-(2-Alkenyl)-7-oxo- 600 (7331)
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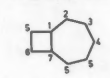

8-Oxo- 600 (7331)
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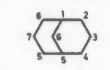

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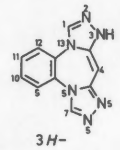

3*H*-

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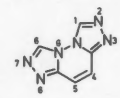

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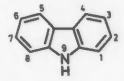

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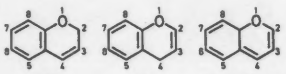

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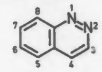


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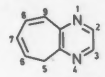

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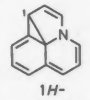

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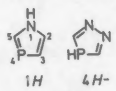

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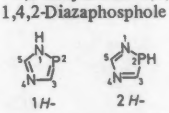

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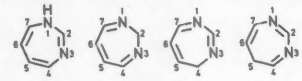

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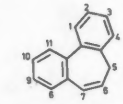

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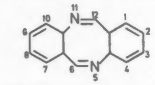

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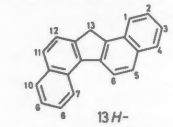

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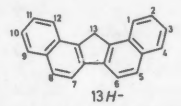

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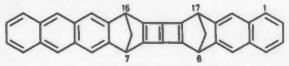

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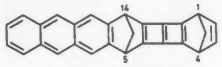

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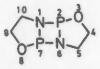

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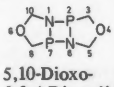

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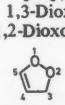

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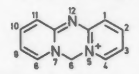

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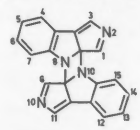

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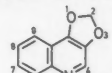

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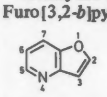

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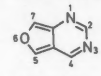

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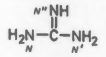

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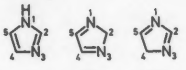

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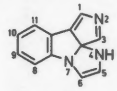

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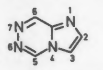

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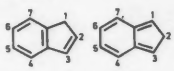

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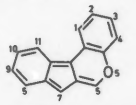

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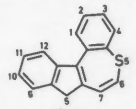

8*H*-

6,7-Dihydro-8*H*-, 8-Alkylthio-8-aryl- 744
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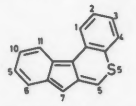

6,7-Dihydro-, 7-Alkylthio-7-aryl- 744
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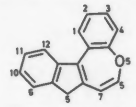

6,7-Dihydro-8*H*-, 8-Alkylthio-8-aryl- 744
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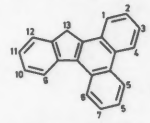

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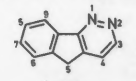

5*H*-

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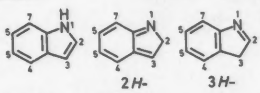

2*H*- 3*H*-

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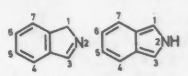

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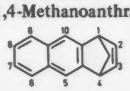

1,4-Dihydro-

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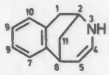

1,2,3,6-Tetrahydro-

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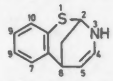
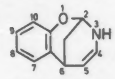


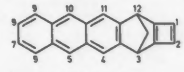
3,6-Dihydro-2*H*-

3,6-Dihydro-2*H*-, poly-substituted 144
2,6-Methano-1,3-benzoxazocine



3,6-Dihydro-2*H*-

3,6-Dihydro-2*H*-, poly-substituted 144
3,12-Methanocyclobuta[*b*]tetracene



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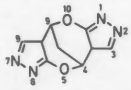

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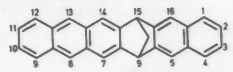

6,15-Dihydro-

6,15-Dihydro- 328
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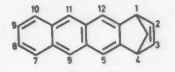

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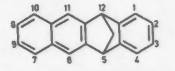

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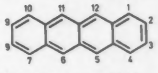

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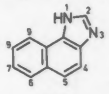
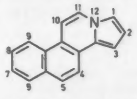


1*H*-

Naphtho[1,2-*g*]indolizine



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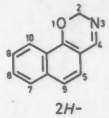

2*H*-

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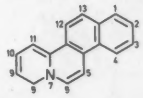

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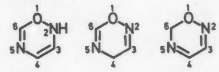

2*H*- 4*H*- 6*H*-

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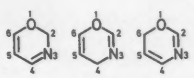

2*H*- 4*H*- 6*H*-

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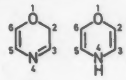

2*H*- 4*H*-

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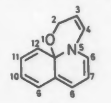

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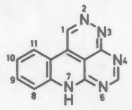

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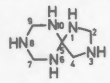

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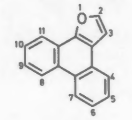

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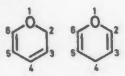

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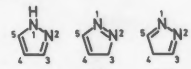

3*H*- 4*H*-

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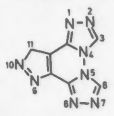
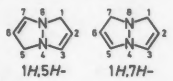


11*H*-

**Pyrazolo[1,2-*a*]pyrazole**



1*H*,5*H*- 1*H*,7*H*-

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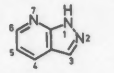

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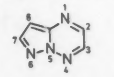
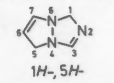


**Pyrazolo[1,2-*a*][1,2,4]triazole**



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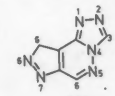

9*H*-

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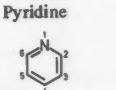

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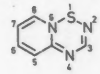

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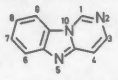

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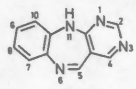

11*H*-

2,3-Dihydro-11*H*-, 5-Amino-2-oxo- 668
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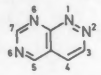

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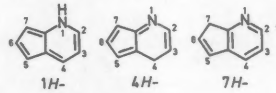

1*H*- 4*H*- 7*H*-

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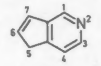

5*H*-

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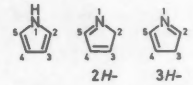

2*H*- 3*H*-

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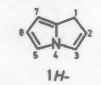

1*H*-

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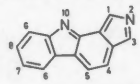

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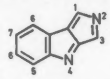

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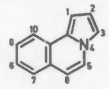

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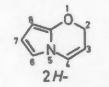

2*H*-

6,7,8,8a-Tetrahydro-2*H*-, 2-Alkoxy- 899
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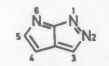

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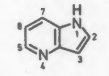

1*H*-

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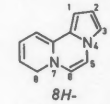

8*H*-

5,6-Dihydro-8*H*-, 8-Dicyanomethylene- 908
5,6,9,10-Tetrahydro-8*H*-, 8-Dicyanomethylene- 908
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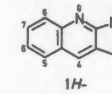

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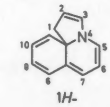

1*H*-

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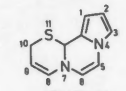

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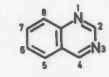

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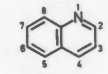

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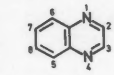

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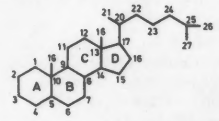

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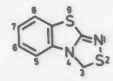
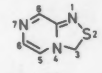
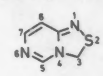


3*H*-
3-Phenylimino-3*H*- 1027
[1,2,4]Thiadiazolo[4,3-*a*]pyrazine



3*H*-
3-Phenylimino-3*H*- 1027
[1,2,4]Thiadiazolo[4,3-*c*]pyrimidine



3*H*-

3-Phenylimino-3*H*-[1,3,4]thiadiazolo[2,3-*c*][1,2,4]thiadiazole 1027

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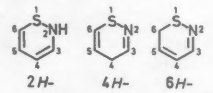

2*H*- 4*H*- 6*H*-

2,3-Dihydro-6*H*-1,2-thiazine 1-Oxides

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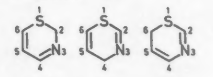
- Synthesis and Cleavage 246 (7264)

1,3-Thiazine



2*H*- 4*H*- 6*H*-

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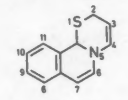
[1,3]Thiazino[2,3-*a*]isoquinoline



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1,3-Thiazoles

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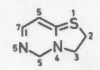
[1,3]Thiazolo[3,2-*c*]pyrimidine



5*H*-

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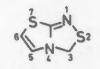
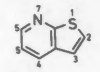
[1,3]Thiazolo[2,3-*c*][1,2,4]thiadiazole



3*H*-

3-Phenylimino-3*H*- 1027

Thieno[2,3-*b*]pyridine



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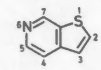

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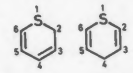

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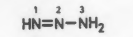

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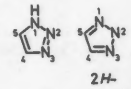

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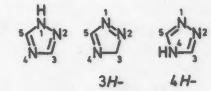

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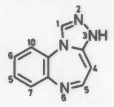

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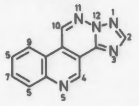

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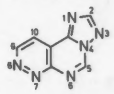

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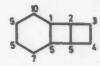

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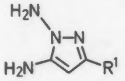
# Errata and Addenda 1986

I. Ganboa, C. Palomo *Synthesis* **1986**, 52. The $^1$H-NMR data for compounds **2d** and **2e** in the Table (p. 53) should be, respectively: 8.13 (d, 2 $H_{arom}$); 7.46 (d, 2 $H_{arom}$); 7.3 (s, 5 $H_{arom}$); 5.73 (m, 1 H, C−H); 5.26 (s, 2 H, $CH_2$−$C_6H_4NO_2$); 4.9 (m, 1 H, C−H); 3.7 (m, 2 H, $CH_2$−CO−NH); 3.3 (m, 2 H, S−$CH_2$); 2.13 (s, 3 H, $CH_3$). 7.33 (s, 5 $H_{arom}$); 7.3 (s, 5 $H_{arom}$); 5.76 (m, 1 H, C−H); 5.2 (s, 2 H, $C_6H_5$−$CH_2$); 4.9 (m, 1 H, C−H); 3.63 (s, 2 H, $CH_2$−CO−NH); 3.3 (m, 2 H, S−$CH_2$); 2.13 (s, 3 H, $CH_3$).

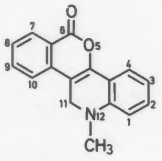
The $^1$H-NMR data for compound **6** (p. 54) should be:
$^1$H-NMR ($CDCl_3$/$TMS_{int}$): $\delta$ = 8.03 (d, 2 $H_{arom}$); 7.43 (d, 2 $H_{arom}$); 5.65 (s, 1 H, CH); 5.23 (s, 2 H, $CH_2$); 4.5 (s, 1 H, NH); 1.53, 1.35 ppm (2 s, 6 H, 2 $CH_3$).

K. Tanaka, H. Yoda, K. Inoue, A. Kaji *Synthesis* **1986**, 66. The $[\alpha]_D^{25}$ value for compound **2e** in Table 1 (p. 67) should be: − 28.2° (1.80).

D. R. Sliskovic, M. Siegel, Y. Lin *Synthesis* **1986**, 71. The structures for compounds **6a, b** (p. 73) should be:

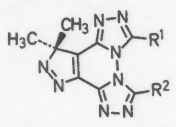


O. Meth-Cohn *Synthesis* **1986**, 76. The correct numbering for compounds **8** and **10** (p. 76) is as illustrated below for compound **10**:



**10**

B. Furlan, B. Stanovnik, M. Tišler *Synthesis* **1986**, 78. The double-bond arrangement of compounds **3**, **6**, and **7** (pp. 78, 79) should be:

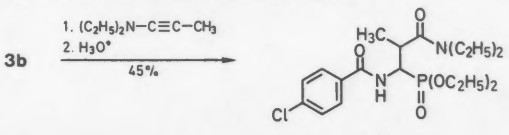


N. Petragnani, H. M. C. Ferraz, G. V. J. Silva *Synthesis* **1986**, 157. The authors wish to include the following pertinent references:
R. M. Adlington, A. G. M. Barret *Tetrahedron* **1981**, *37*, 3935.
R. M. Adlington, A. G. M. Barret *J. Chem. Soc. Perkin Trans. 1* **1981**, 2848.
R. M. Adlington, A. G. M. Barret *J. Chem. Soc. Chem. Commun.* **1981**, 65.
R. M. Adlington, A. G. M. Barret *J. Chem. Soc. Chem. Commun.* **1979**, 1122.

A. J. Fatiadi *Synthesis* **1986**, 249. The heading for the first experimental procedure on p. 268 should be:
**2,6-Diphenyl-4-(2,3,3-tricyanoallylidene)pyran (201)[354]:**

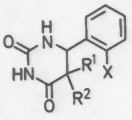
D. P. Matthews, J. P. Whitten, J. R. McCarthy *Synthesis* **1986**, 336. The headings for the first and last experimental procedures should be, respectively:
***N*$^1$,*N*$^3$-Bis(2,2-dimethoxyethyl)oxaldiamidine Dihydrochloride (2):**
**2-(2-Imidazolyl)-4-methoxy-4,5-dihydroimidazole (5):**

T. Schrader, R. Kober, W. Steglich *Synthesis* **1986**, 372. The last equation in the formula scheme (p. 372) should be:

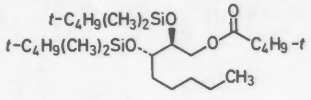


**6**

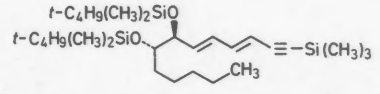
D. N. Dhar, K. S. K. Murthy *Synthesis* **1986**, 437. The heading for Table 2 (p. 440) should be:
4-Aryl-2(1*H*)-quinazolines (**13**) and 4-Aryl-1*H*-2,1,3-benzothiadiazine 2,2-Dioxides (**14**)
The names of compounds **13a** and **14a** in the experimental procedure on the same page should be corrected accordingly.
For compounds **60** and **61** (p. 445) $R^3$ = H, $SO_2Cl$.
The product in the lower, left reaction scheme on p. 446 should be:



K. C. Nicolaou, S. E. Webber *Synthesis* **1986**, 453. The structures of compounds **8** (p. 454) and **16** (p. 455) should be:



**8**

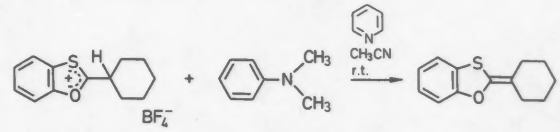


**16**

E. Dalcanale, M. Foà *Synthesis* **1986**, 492. In the reaction scheme, products **4** and **5** are obtained in 33 and 8%, respectively, a ratio of 80:20.

W. G. Dauben, J. M. Gerdes, G. C. Look *Synthesis* **1986**, 532. In the experimental procedure headings (p. 534), the names of compounds 3, 5, 7, and 9 should read:
**(3,3-Ethylenedioxybutyl)triphenylphosphonium Bromide (3)**
**6-*t*-Butyldimethylsiloxy-3,7-dimethyl-1,6-octadiene (5)**
**5-[1,1-Bis(ethoxycarbonyl)ethyl]bicyclo[3.3.0]octan-2-one (7)**
**2,2-Ethylenedioxy-1,3,3-trimethylbicyclo[2.2.1]heptane (9).**

S. Cadamuro, I. Degani, R. Fochi, A. Gatti, V. Regondi *Synthesis* **1986**, 544. Formula Scheme **B** should be:



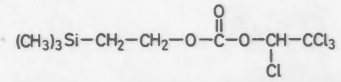
**1c** **2** **5**

H. M. R. Hoffmann, K. Giesel, R. Lies, Z. M. Ismail *Synthesis* **1986**, 548. The heading for the last experimental procedure (p. 551) should be:
**Cycloadditions; 4-Oxatricyclo[7.2.1.0$^{2,8}$]dodeca-3,10-dien-2-one (11e):**

Abstract 7330, *Synthesis* **1986**, 599. The structure of compound **7** should be: $CH_2$=C($R^6$)$R^7$.

Abstract 7333, *Synthesis* **1986**, 600. Line 2 of the text should read: dimenthyl succinate (**1**) with lithium 2,2,6,6-tetramethylpiperidide reacts...

G. Barcelo, J. P. Senet, G. Sennyey, J. Bensoam, A. Loffet *Synthesis* **1986**, 627. The structure of compound **1k** (p. 630) should be:

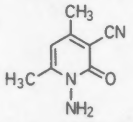


D. Achet, D. Rocrelle, I. Murengezi, M. Delmas, A. Gaset *Synthesis* **1986**, 642. The last word of the title should be: **Sulfate**

H. Herzog, H.D. Scharf *Synthesis* **1986**, 788. The heading for the experimental procedure on p. 789 (top, right) should be:
*Step E:* **(1*R*,2*S*,4*S*)-2-benzyloxybornan-3-one (8):**

A.N. Pudovick, I.V. Konovalova, L.A. Burnaeva *Synthesis* **1986**, 793. The text starting in line 2 on page 798 (top, right) should read: leads to 3,3a-dihydro-4*H*-1,2-azaphospholo[5,4-*b*]pyridines **41**[1,63].

R.C. Phadke, D.W. Rangnekar *Synthesis* **1986**, 860. The structure of compound **1** (p. 861) should be:




B. Byrne, K.J. Wengenroth *Synthesis* **1986**, 870. The heading for the first experimental procedure should be:
**2-(1-Bromoethyl)-2-ethyl-1,3-dioxolane (1):**

S. Torii *Synthesis* **1986**, 873. The heading for the experimental procedure on p. 882 should be:
**Electrosynthesis of 1-Benzyl-6,9-dimethyl-2,5-dioxo-8-tosylamino-1,2,3,4,5,6-hexahydro-1-benzazocine (81):**

N.G. Bushmakina, A.Y. Misharan *Synthesis* **1986**, 966. The heading for the first experimental procedure should be:
**2,2,6,6-Tetramethyl-4-methylsulfonyloxy-1-piperidinyloxy Radical (3):**

X. Huang, B.C. Chen *Synthesis* **1986**, 967. The title should be:
**Synthesis of Bis(alkylthio)methylene Derivatives of Meldrum's Acid and Barbituric Acid**
Throughout the paper the expression "bisalkylthioylide" should be replaced by "bis(alkylthio)methylene".
The heading for the second experimental procedure should be:
**5-(1,3-Dithian-2-ylidene)barbituric Acid (6d); Typical Procedure:**

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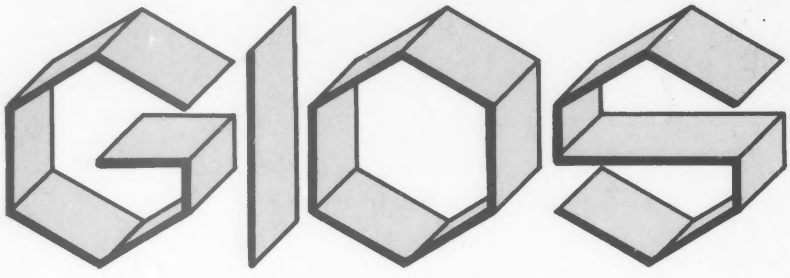


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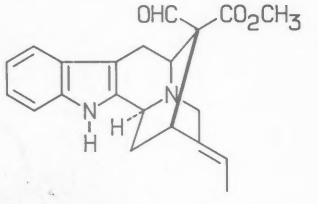
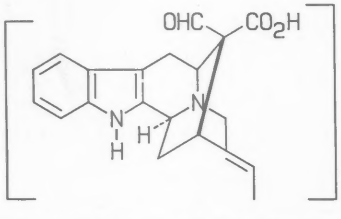


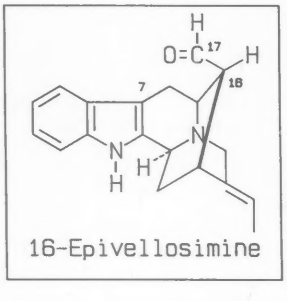

Polyneuridine aldehyde

PNA-esterase




Polyneuridine aldehyde acid

spontaneous

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